Measurement 125 (2018) 257–261

Contents lists available at ScienceDirect

Measurement

journal homepage: www.elsevier.com/locate/measurement

An improved noninvasive resonance method for water content characterization of Cultural Heritage stone materials

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ARTICLE INFO

Keywords:
Cultural Heritage
Dielectric permittivity
Moisture content measurements
Patch resonator
Stones

ABSTRACT

In this work, a noninvasive microwave-based system for monitoring water content of Cultural Heritage stone materials is presented. In particular, by placing a planar resonator in contact with the stone sample, an experimental relationship between resonant frequency and water content is obtained.

To verify the suitability of the system, experimental tests are carried out on several types of stones: gentile; leccese; carparo; red brick; and red brick fabricated at high temperatures. The first three types of stones are typically found in Cultural Heritage structures, and they are particularly affected by deterioration and decay phenomena. As for the red bricks, they are found in buildings from the XVII Century.

Measurements are performed for five levels of water content of the stone samples, and the empirical relationship between each considered water content level and the corresponding resonance frequency of the patch resonator are derived. To enhance the contact between the planar resonator and the rough surface of the stone, the patch is covered with a thin layer of soft silicon conductor. The obtained results demonstrate the robustness of the presented solution.

1. Introduction

The monitoring and preservation of Cultural Heritage is a heartfelt issue not only for the Scientific Community, but also for the general population, thanks to the widespread awareness of the importance of preserving the invaluable cultural treasures.

Because moisture is one of the major causes of decay of ancient building materials, a wide range of measurement techniques is applied for detecting moisture presence. Among these, nuclear magnetic resonance (NMR) portable devices can be used for nondestructive in situ analysis of water content in Cultural Heritage structures [1,2]. In particular, when using mobile stray-field NMR, samples need not be placed within the magnet but can be examined externally in the stray magnetic field [3]. Also, neutron scattering and neutron imaging are powerful techniques for studying the structure of objects without damage [4,5]. Furthermore, ground penetrating radar (GPR) [6–8] and spectroscopic techniques [9] have been extensively used for non-invasive moisture characterization of subsurfaces. Finally, infrared thermography is another well-established technique that is employed for the evaluation of water transport phenomena through the outer layers of porous stone materials [10].

Indeed, to obtain more comprehensive results, different techniques are often used in combination. For example, in [11], the combination of microwave transmission, spectral-induced polarization, and laser-induced breakdown spectroscopy was employed for assessing moisture and salt contents in brick masonry. Evanescent field dielectrometry (EFD), unilateral NMR and holographic radar were complementarily used in [12] to measure the water present in a wall.

In [13,14], to integrate the GPR survey with complementary data and enhance data interpretation, also electrical resistivity tomography (ERT) profiles are used.

However, the use of the aforementioned techniques (used alone or in combination) requires the operator to possess in-depth technical/scientific knowledge. On the other hand, microwave reflectometry-based measurement systems allow a noninvasive approach and adequate accuracy; additionally, they are also easy to be used in the field, and they can be implemented in low-cost versions. In [15], different microwave-based methods and probes were comparatively used to infer, noninvasively, the relationship between water content θ of stone materials and the reflection properties at microwave frequency of the material. More specifically, three types of probes (an open-ended coaxial probe; a patch resonator; and an open-ended waveguide) and

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https://doi.org/10.1016/j.measurement.2018.04.070
Received 6 March 2018; Received in revised form 17 April 2018; Accepted 19 April 2018

two different measurement instruments (a vector network analyzer and a time-domain reflectometer) were employed for the characterization of stones used in Cultural Heritage. The obtained results demonstrated that the use of a patch resonator as a probe can provide several advantages over the other considered methods; in particular, by placing the stone sample in contact with the planar resonator, the variation of the resonance frequency of the resonator can be related to the water content of the stone sample.

Starting from these considerations, in this work, experimental tests are carried out on several types of materials: *gentile*; *leccese*; *carparo*; and red bricks (fabricated at different temperatures). The first three types of stones are typically found in Cultural Heritage structures, and they are particularly affected by deterioration and decay phenomena. As for the red bricks, they are found in buildings from the XVII Century.

In order to circumvent the problem related to the roughness of the stone surfaces (which may compromise the adherence between the patch and the sample surface), the patch was covered with an electrically-conductive silicone, which could adapt to the rough surface of the stones.

Measurements were performed for increasing level of water content of the stone samples, and the empirical relationship between each considered level of water content and the corresponding measured resonance frequency was derived.

2. Background

The basic principle is to exploit the fact that the presence of water, whose relative dielectric permittivity is in the order of 78 [16], increases the dielectric permittivity of the considered stone materials (which, in dry conditions, exhibit a relative dielectric permittivity of the order of 5–6).

As for the resonant frequency (f_r) of a patch resonator, it depends not only on the permittivity of the substrate, but also on the permittivity of the medium in which the resonator radiates [17,18]:

$$f_r = \frac{c}{\sqrt{\varepsilon_{eff}}\cdot 2L_{eff}}, \tag{1}$$

where L_{eff} is the effective length of the radiating patch and ε_{eff} is the effective relative permittivity. Both these quantities depend on the relative dielectric permittivity of the material in which the antenna radiates.

Starting from these considerations, the idea is to place the planar resonator in contact with the considered stone sample, and to measure the corresponding f_r value, thus obtaining an experimental relationship between f_r and the water content value.

3. Materials and methods

3.1. Materials

Four types of stones were considered: *gentile*; *carparo*; red brick; and *leccese*.

Gentile stone is a calcarenite ground stone, and its mineralogical composition is that of calcite [19]. Thanks to its good workability properties, this stone has been widely used in the building sector with several functions, for ashlars and load-bearing elements but also for coatings, decorations and statuary [20].

As for *carparo* stone, this is a limestone from the South of Italy (particularly used in the Salento area). Its rough appearance derives from cementation of sediments of limestone, in the marine environment [21]. This material is commonly used in construction stone, ornamental stone, and architecture. It was especially employed in the Baroque era, for the facades of several churches and historic buildings; an example is the wonderful cathedral of Sant'Agata in Gallipoli (Lecce, Italy). Being a natural material, *carparo* does not have a completely homogeneous appearance and may vary in grain size and color gradation depending on the concentration of chemical components and the different extraction points.

Table 1
Dimensions of the stone samples.

Type	Dimensions (cm × cm × cm)
gentile stone	19.8 × 19.9 × 2.3
carparo stone	19.9 × 19.7 × 2.1
leccese stone	19.8× 19.9 × 2.0
red brick stone	22.2 × 11.0 × 5.3
red brick-HT stone	21.8 × 10.9 × 3.0

Leccese stone has been used, since ancient times, to construct private and public buildings, and as ornamental stone [22]. It has strong aptitude towards absorbing and retaining water; unfortunately, its porosity makes it prone to degradation.

Red brick stones consist mainly of baked clay and sand, and many of their characteristics vary with the type of clay and firing process. Bricks range in texture from very even and smooth pressed faces to very rough rustic faces [23]. In particular, fired bricks are burned in a kiln which makes them durable. Well maintained brickwork and pointing should not enable water penetration and should require no additional external coatings to prevent water penetration. However, if the outer surface has been removed, for example by sandblasting, water may start to infiltrate.

The stone samples were cut with the dimensions reported in Table 1.

3.2. Methods

As mentioned in Section 3.1, the presented measurement system relies on establishing the empirical relationship between the resonant frequency (f_r) of a patch resonator and the water content (θ) of the stone sample, when the planar resonator is placed on the sample [24], as shown in Fig. 1(a). For this purpose, a patch resonator was designed and fabricated [24].

To circumvent the problem of the non-perfect contact between the copper patch and the stone samples (which were not polished), a silicon conductor with the same size of the patch and thickness 1.7 mm was placed on the antenna patch, as shown in Fig. 1(b). As shown in the figure, the silicon was attached to the patch through adhesive tape; however, in practical applications, electrically conductive glue may be used in place of the tape.

The reflection scattering parameter of the resonator, $S_{11}(f)$, was measured by placing the resonator in contact with the stone samples (moistened at different reference values of θ_{ref}). For each value of θ_{ref}, the $S_{11}(f)$ was measured through a vector network analyzer (VNA, model Agilent E8363C). From the magnitude of the $S_{11}(f)$, the corresponding f_r value was inferred. Finally, the measured f_r values were associated to the corresponding θ_{ref} value.

For each stone sample, to bring the considered samples to reference (known) water content values, θ_{ref}, to be used for testing the measurement systems, the following moistening procedure was followed:

(1) drying of the sample in a microwave oven;
(2) weighing of the dry sample, W_{dry};
(3) bath in deionized water until saturation (which, for the considered samples, took less than two hours);
(4) weighing of the sample, W_i;
(5) assessment of the volumetric water content, θ_{ref}:

$$\theta_{ref} = \frac{(W_i - W_{dry})}{V_{stone}\cdot\rho_w}\times 100 \tag{2}$$

where V_{stone} is the volume of the stone sample, and $\rho_w \cong 0.996$ g/cm^3 is the density of water.

(6) oven drying for a limited amount of time, in order to remove part of the moisture.